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IS : 1448 [P : 22] - 1985
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Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 22]

ASPHALTENES PRECIPITATION WITH NORMAL HEPTANE

(Second Revision)

First Reprint OCTOBER 2007
(Including Amendment No. 1)

UDC 665.6/.7 : 543 : 549.883

Adapted with permission from the Institute of Petroleum Standard No. IP 143/82

1. SCOPE

1.1 This method is applicable to gas oil, diesel fuel, fuel oil, lubricating oil, bitumen, and to crude petroleum which has been topped to an oil temperature of 260°C. Oils containing additives may give erroneous results.

2. TERMINOLOGY

2.0 For the purpose of this method, the following definition shall apply.

2.1 Asphaltenes Content – The percentage by mass of wax-free material insoluble in *n*-heptane but soluble in hot benzene. The term *asphaltenes* is synonymous with *hard asphalt content.*

3. OUTLINE OF THE METHOD

3.1 The material is dissolved in *n*-heptane and the insoluble material, consisting of asphaltenes and waxy substances, is separated by filtration

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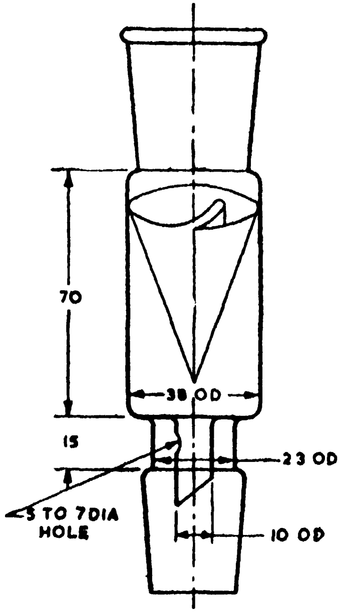
through a fine filter paper. The waxy constituents are extracted under hot reflux with *n*-heptane, and the asphaltenes are isolated by extraction with toluene or benzene.

3.2 The extraction with benzene is not normally recommended because of the high toxicity of this material and should only be used for referee purpose.

3.3 In normal practice use toluene instead of benzene. The precision of the method when using toluene has been found to be the same as when using benzene.

4. APPARATUS

4.1 Extraction Apparatus — consisting of an efficient form of condenser, for example, with a coil or double surface, a reflux extractor of the type as given in Fig. 1, and a conical flask. Ground-glass joint shall be used throughout. The dimensions of the extractor as shown in Fig. 1 are critical and shall be adhered to.



All dimensions in millimetres.

FIG. 1 EXTRACTION APPARATUS

4.2 Evaporating Dish — a hemi-spherical dish of heat-resistant glass, having a diameter of approximately 90 mm.

5. REAGENTS

5.1 *n*-Heptane — conforming to the following requirements:

Density at 20°C, g/ml	0·683 65 to 0·683 95
Refractive index at 20°C	1·387 55 to 1·387 85
Freezing point, *Min*	−90·710°C
Distillation:	
a) Temperature of 50 percent recovery	98·402 to 98 452°C
b) Temperature increase from 20 to 80 percent recovery, *Max*	0·020°C

5.2 Toluene — *See* IS : 1839-1961[*].

CAUTION — Toluene is a toxic, volatile hydrocarbon which is absorbed by inhaling the vapour or through the skin by contact with the liquid. Use in adequate ventilation and avoid skin contact.

5.3 Benzene — *See* IS : 1840-1961[†].

CAUTION — Benzene is a highly toxic, volatile hydrocarbon which is absorbed by inhaling the vapour or through the skin by contact with the liquid. Use under extraction ventilation, avoid skin contact and wear approved protective gloves.

6. SAMPLE

6.1 For all materials except crude oils proceed as described in **8**; for crude oils prepare a residue by distilling 90 to 100 ml of the sample with the bulb of the thermometer in the oil and stopping the distillation at an oil-temperature of 260°C. Calculate and record the mass of the residue as percentage of the original sample.

6.2 If the sample is hard bitumen, grind it to a powder immediately before the test.

7. PREPARATION OF APPARATUS

7.1 Clean all glass-dishes by immersing them in concentrated sulphuric acid (*see* IS : 266-1977[‡]) or chromic acid mixture for at least 12 hours. Rinse with distilled water and with acetone. Place in an oven at 100 to

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[*] Specification for toluene, reagent grade.

[†] Specification for benzene, reagent grade.

[‡] Specification for sulphuric acid (*second revision*).

110°C for 30 minutes, and cool in a desiccator for 30 minutes before weighing.

> *CAUTION* — Chromic acid is potentially hazardous in contact with organic materials and is toxic and highly corrosive. Sulphuric acid is also highly corrosive. If either are used, wear approved full face-shield and full-length protective clothing including gloves.

8. PROCEDURE

8.1 Into a conical flask of suitable capacity weigh to the nearest 0·01 g sample, or of the residue obtained in **6**, not exceeding 10 g which will give a final mass of asphaltenes not exceeding 0·25 grams. Add *n*-heptane in the ratio of 30 ml to each 1 g of sample, and boil the mixture under reflux for one hour. When the asphaltene content of the sample exceeds 25 percent, the volume of *n*-heptane taken is small and there is danger of bumping during refluxing. In such cases it is permissible to increase the volume *n*-heptane to a maximum dilution ratio of 50 : 1. Bumping is less likely to occur in small flasks. Remove the flask and contents from reflux, cool, close with ground-glass stopper, and store in a dark cupboard for one and a half hours to two and a half hours calculated from the time of removal from reflux.

8.2 Without agitation, decant the liquid through a Whatman (No. 42) filter paper of fine porosity, 11·0 or 12·5 cm diameter, folded as shown in Fig. 2 so as to prevent loss of asphaltenes by creeping. Transfer the residue in the flask as completely as possible to the filter with successive quantities of hot *n*-heptane, using a glass rod if necessary. Give the flask a final rinse with hot *n*-heptane, and pour the rinsings through the filter. Set the flask aside for further use, without washing, as prescribed in **8.4**.

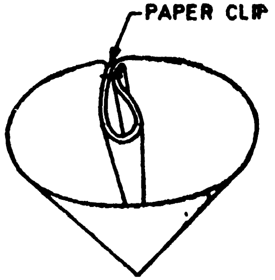


Fig. 2 Folded Filter Paper

8.3 Remove the filter-paper and contents from the funnel and place in an extractor of the type illustrated in Fig. 1. Using a flask different from that used initially, reflux with *n*-heptane at a rate of 2 to 4 drops/seconds from the end of the condenser for an extraction period of not less than one hour or until a few drops of *n*-heptane from the bottom of the extractor leave no residue on evaporation on a glass slide.

8.4 Replace the flask by the one used initially and to which has been added 30 to 60 ml of toluene/benzene and continue refluxing until all the asphaltenes have been dissolved from the paper.

8.5 Transfer the contents of the flask to a glass evaporating dish which has been cleaned and dried as described in 7 and weighed, to the nearest 0·2 mg, by tare against a similar dish. Wash out the flask with successive small quantities of toluene/benzene to a total not exceeding 30 millilitre. Remove the toluene/benzene by evaporation on a boiling water-bath.

> *CAUTION* — Perform this operation in a fume cupboard. Dry the dish and contents by heating in an oven at 100 to 110°C for 30 minutes (*see* Note). Cool in a desiccator for half an hour to one hour and reweigh by tare against the dish used previously for this purpose, and which has been subjected to the same heating and cooling procedure as the dish containing asphaltenes.

NOTE — Asphaltenes are very susceptible to oxidation, and it is important that the procedure specified in the final drying stage shall be adhered to strictly as regards temperature and time.

9. CALCULATION AND REPORTING

9.1 Calculate the asphaltenes content as a percentage by mass of the original sample taken for test and report the results as indicated below:

a) Values less than 0·05 percent as 'Less than 0·05 percent by mass',

b) Values of 0·05 percent and higher, but less than 1·0 percent, to two decimal places; and

c) Values of 1·0 percent and higher, to the nearest 0·1 percent.

10. PRECISION

10.1 Results of duplicate tests should not differ by more than the following amount:

Repeatability	*Reproducibility*
10 percent	20 percent

10.2 For values less than 0·5 percent, the method may be less precise than indicated.

AMENDMENT NO. 1 FEBRUARY 1995
TO
IS 1448 [P : 22] : 1985 METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

PART 22 ASPHALTENES PRECIPITATION WITH NORMAL HEPTANE

(Second Revision)

(*Page* 1, *clause* **1.1**) — Add the following after **1.1**:

'**1.2** This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.'

(*Page* 3, *clause* **6.1**) — Add the following after **6.1**:

'(*See also* Annex A)'.

(*Page* 5, *clause* **10.2**) — Add the following after **10.2**:

'ANNEX A

(*Clause* **6.1**)

(This Annex forms an integral part of the test method)

PREPARATION OF CRUDE PETROLEUM RESIDUE BY DISTILLATION

A-1 GENERAL

This Annex describes a procedure for the preparation of a crude petroleum residue suitable for the determination of asphaltene content.

A-2 PRINCIPLE

A weighed portion of the sample is distilled in specified glass apparatus under prescribed conditions of heat input and rate of distillation. The distillation is stopped at an oil temperature of 260°C and the mass of the residue determined.

A-3 APPARATUS

A-3.1 Distillation Equipment — A typical assembly of the apparatus is shown in Fig. 3 and 4.

A-3.1.1 *Distillation Flasks*

Flasks of heat-resistant glass shall be provided. Construction details and tolerances for the flask are given in Fig. 5. Flasks with bulb diameter at the minimum specification can cause problems with some liquids due to liquid contact with thermometer area before the IBP.

A-3.1.2 *Condenser and Cooling Bath*

A-3.1.2.1 Typical approved types of condenser and cooling bath are included in Fig. 3 and 4.

A-3.1.2.2 The condenser shall be made of seamless brass tubing, 560 mm in length. It shall be 14 mm in outside diameter, and shall have a wall thickness of 0.79 to 0.91 mm.

A-3.1.2.3 The condenser shall be set so that approximately 390 mm of the tube will be in contact with the cooling medium, with about 114 mm outside at the lower end. The length of tube projecting at the upper end shall be straight and shall be set at an angle of 75 deg with the vertical. The section of the tube inside the cooling bath may be either straight or bent in any suitable continuous, smooth curve. The average gradient shall be 0.26 mm per linear mm of condenser tube (sine of angle of 15 deg), and no section of the immersed portion of the condenser tube shall have a gradient less than 0.24 and not more than 0.28 mm per linear mm of tube. The projecting lower portion of the condenser tube shall be curved downward for a length of 76 mm and slightly backward so as to insure contact with the wall of the receiving graduate at a point approximately 25-32 mm below the top of the graduate when it is in position to receive the distillate. The lower end of the condenser tube shall be cut off at an acute angle so that the tip may be brought into contact with the wall of the cylinder.

A-3.1.2.4 The capacity of the cooling bath shall be not less than 5.55 litres of cooling medium. The arrangement of the tube in the cooling bath shall be such that its centre line shall be not less than 32 mm below the plane of the top of the bath at its point of entrance and not less than 19.0 mm above the floor of the bath at its exit.

A-3.1.2.5 Clearances between the condenser tube and the walls of the bath shall be atleast 12.7 mm except for the sections adjacent to the points of entrance and exit. Multiple installations are permissible, provided they conform to the dimensional requirements and the capacity of the bath is not less than 5.55 litres per tube.

A-3.1.3 *Metal Shield or Enclosure for Flask*

A-3.1.3.1 Type 1 Shield (Fig. 3) is 480 mm high, 280 mm long and 200 mm wide, made of sheet metal of approximately 0.8 mm. It shall have a door on one narrow side, and two openings 25 mm in diameter, equally spaced in each of the two narrow sides, with a slot cut in one side for the vapour tube. The centres of these four openings shall be 216 mm below the top of the shield. There shall be three 12.7 mm holes in each of the four sides, with their centres 25 mm above the base of the shield.

A-3.1.3.2 Type 2 Shield (Fig. 4) is 440 mm high, 200 mm long, and 200 mm wide, made of sheet metal of approximately 0.8 mm, with a window on the front side. The open bottom of the shield shall be spaced approximately 50 mm from the base of the unit. The rear of the shield shall have an elliptical hole for the vapour tube. A flask-adjusting knob shall be located on the front of the shield for adjusting the flask support. Also, a heat-adjusting indicating dial shall be used to provide stepless heat control when the electric heater is used. The entire mechanism shall be built into the bottom portion of the shield. When the electric heater is employed, the portion of the shield above the board shall be the same as with the gas burner, but the part below may be omitted provided this does not cause the distillation flask to be exposed to draughts.

A-3.1.4 *Heat Source*

A-3.1.4.1 Gas Burner (Fig. 3), so constructed that sufficient heat from the available gas can be obtained to distil the product at the specified rate. A sensitive regulating valve and gas pressure governor to give complete control of heating may be provided.

A-3.1.4.2 Electric Heater (Fig. 4), may be used instead of a gas burner, provided it is capable of bringing over the first drop from a cold start within the time specified and of continuing the distillation at the specified rate. Heater units of low heat retention, adjustable from 0-1 000 W, have been found satisfac- tory.

A-3.1.5 *Flask Support*

A-3.1.5.1 *Type 1 for use with gas burner* (Fig. 3) — A ring support of the ordinary laboratory type, 100 mm or larger in diameter, supported on a stand inside the shield, or a platform adjustable from the outside of the shield, may be used.

Two ceramic heat resistant boards, 3 to 7 mm in thickness, shall rest upon the ring of the platform, whichever is used. The board immediately above the ring or platform shall have a centred opening 76 to 100 mm in diameter and outside line dimensions slightly smaller than the inside boundaries of the shield.

The second or flask support board shall be slightly smaller in outside dimensions than the first board and shall have a 37.5 mm or a 50 mm centred openings. It shall be 3 to 6 mm in thickness at the centre hole rim. This flask support board may be moved slightly in accordance with the directions for placing the distillation flask, and direct heat shall be applied to the flask only through the opening in this board.

A-3.1.5.2 *Type 2 for use with electric heater* (Fig. 4) — The top of the electric heater shall consist centred hole as specified, and a thickness of 3 to 6 mm at the centre hole rim. Provision shall be made for moving the heater unit, with its top, in order to place the distillation flask so that direct heat shall be applied to the flask only through the opening in the flask support board.

A-3.1.6 *Graduated Cylinder*

A 100-ml graduated cylinder with 1 ml sub-divisions shall be provided. Construction details and tolerances are given in Fig. 6.

A-3.2 Thermometer — conforming to the specification for IP 4C or its equivalent.

A-3.3 Drying Apparatus — one of the following.

A-3.3.1 *Centrifuge*

A-3.3.2 *Filter Apparatus* — (increased pressure).

A-3.3.3 *Steel Container-heated.*

A-3.3.4 *Cottrell Type Electrostatic Separator*

A-3.4 Distillation Apparatus — as specified in **A-3.1** but with a 500 ml distillation flask and provided with a transparent bath controlled at 15± 3°C for the receiver.

A-3.5 Separating Funnel — 200 ml capacity.

A-3.6 Graduated Cylinder — 500 ml capacity.

A-4 SAMPLE PREPARATION

A-4.1 If the crude petroleum contains so much water as to make the distillation difficult, dry the sample by any suitable method which avoids the loss of volatile components. The following methods are suitable.

A-4.1.1 Separate the water by gravity or by centrifuging in a closed container at the lowest practicable temperature.

A-4.1.2 Filter the samples at the lowest practicable tempera ture, at atmospheric or increased pressure, through anhydrous calcium chloride, sodium sulphate, or some other suitable drying agent, in a closed vessel.

A-4.1.3 Heat the sample in a closed steel container fitted with a thermometer and pressure gauge. Fill the container to about 70% capacity and heat it until the oil reaches a temperature of 200°C, or until a pressure of 700 kPa is recorded. Allow the container to cool and then decant the oil from the separated water.

A-4.1.4 Separate the water electrostatically by an adaptation of the Cottrell principle. A suitable apparatus consists of a tall glass beaker with a brass gauze cylinder fitting tightly inside and lined with flannel which has been saturated with water and then squeezed out to leave it damp. The central electrode is of brass gauze rolled into a cylinder, mounted on the glass spindle of a laboratory stirrer rotated at about 30 rev/min. Pour the sample into the beaker and apply a voltage to the electrodes from a 12-mm induction coil or some such source. Interrupt the precipitation at intervals to prevent the loss of volatile com ponents and allow the oil to cool. The water globules tend to coalesce and run down the flannel lining.

A-4.1.5 Distil off the water with the more volatile hydrocabrons and separating the water from the distillate, return the remain ing oily distillate to the residue. A suitable apparatus consists of a 500 mL distillation flask connected with a condenser (such as is described in clause A-3.5) through which ice water is circulating; the outlet of the condenser is connected to a separating funnel of approximately 200 ml capacity, immersed in broken ice. Charge 300 ml of the sample into the flask and close it with a cork carrying an IP4C thermometer, so that the bulb of the thermometer is immersed in the sample. Heat the flask gently so that the oil temperature rises slowly to 150°C, vaporising into the condenser by means of a small gas flame any drops of water which collect on the walls of the flask. Stop the distillation when no more water passes over in the distillate and allow the residue to cool to room temperature. Run off the water collected in the separating funnel, return the oily distillate to the flask and mix it thoroughly with the residue. If necessary, the mixture may be dehydrated further by means of calcium chloride.

A-5 PROCEDURE

A-5.1 Determine the density of the oil at 15°C in accordance with IS 1448 (P : 16) : 1990.

A-5.2 Fit the IP4C thermometer tightly into the distillation flask by means of a cork, and adjust it so that the bottom of the bulb is 2±0.5 mm from the bottom of the flask. Remove the thermometer and cork taking care to retain or mark the position of the thermometer in the cork. Weigh the flask to 0.1 g and charge it with the mass of 100 ml of sample at 15°C, to within 0.1 g. Do not allow any of the oil to flow into the vapour tube during this operation. Replace the cork and thermometer.

A-5.3 Swab out the condenser tube to remove any material remaining from a previous test. Place the flask and its contents in position with the vapour tube inserted into the condenser tube. Make this connection tight by means of a stopper or bung through which the vapour tube passes, and adjust the position of the flask so that the vapour tube extends into the condenser tube not less than 25 mm and not more than 50 mm.

A-5.4 Place a clean, dry receiver at the outlet of the condenser so that the condenser tube or the adaptor extends into it atleast 25 mm but not below the 100 ml mark. Immerse the receiver up to the 100 ml mark in the transparent bath of water maintained at 15±3°C. Cover the top of the receiver closely during the distil lation with a piece of paper cut to fit the condenser tube tightly; when necessary use a lead washer or some other device to hold the paper tightly against the top of the receiver and also to provide the additional weight for overcoming the buoyant effect of the liquid in the cooling bath. Circulate ice-cold water through the condenser jacket.

A-5.5 Apply heat to the flask and distil the oil at a rate not exceeding 2.5 ml/min until it ceases to foam, after which keep the rate of distillation constant at 2.0-2.5 ml/min (approximately one drop/s). Continue the distillation without interruption to 260°C. Stop heating the flask and allow the condenser to drain into the receiver.

A-5.6 Allow the residue in the flask to cool, and then weigh the flask and its contents. Calculate and record the mass of the residue as a percentage of the original sample charge.

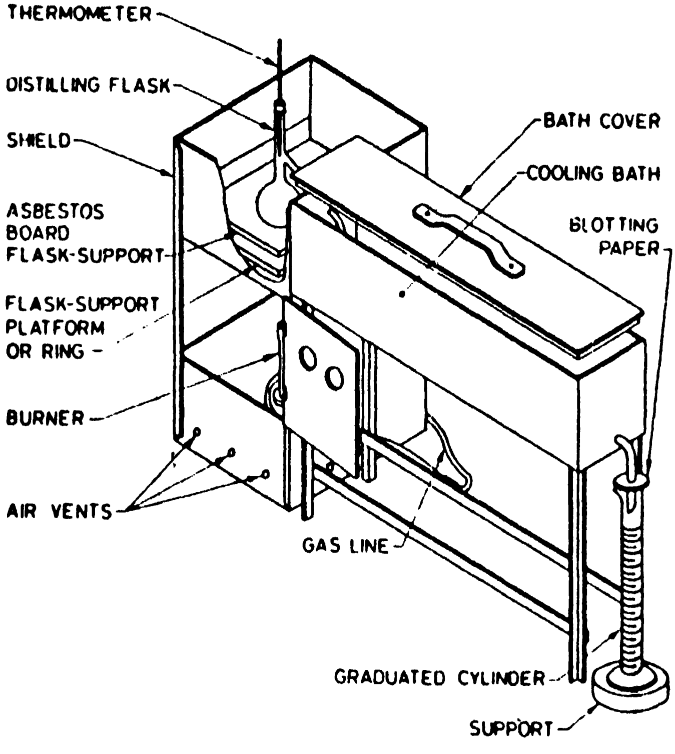


FIG. 3 APPARATUS ASSEMBLY USING GAS BURNER

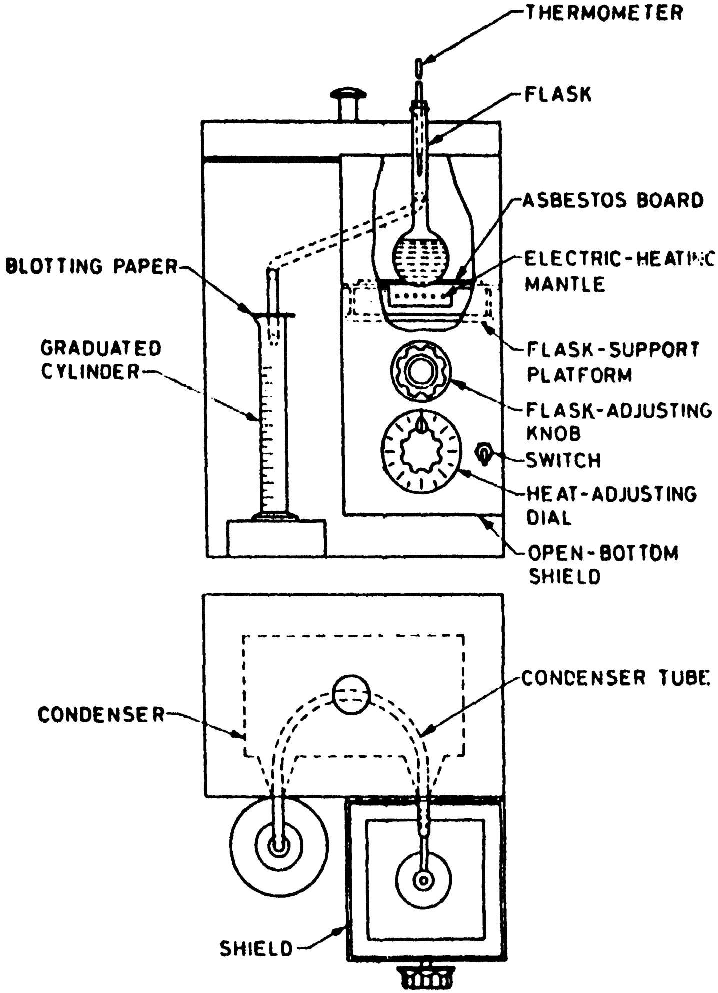


FIG. 4 APPARATUS ASSEMBLY USING ELECTRIC HEATER

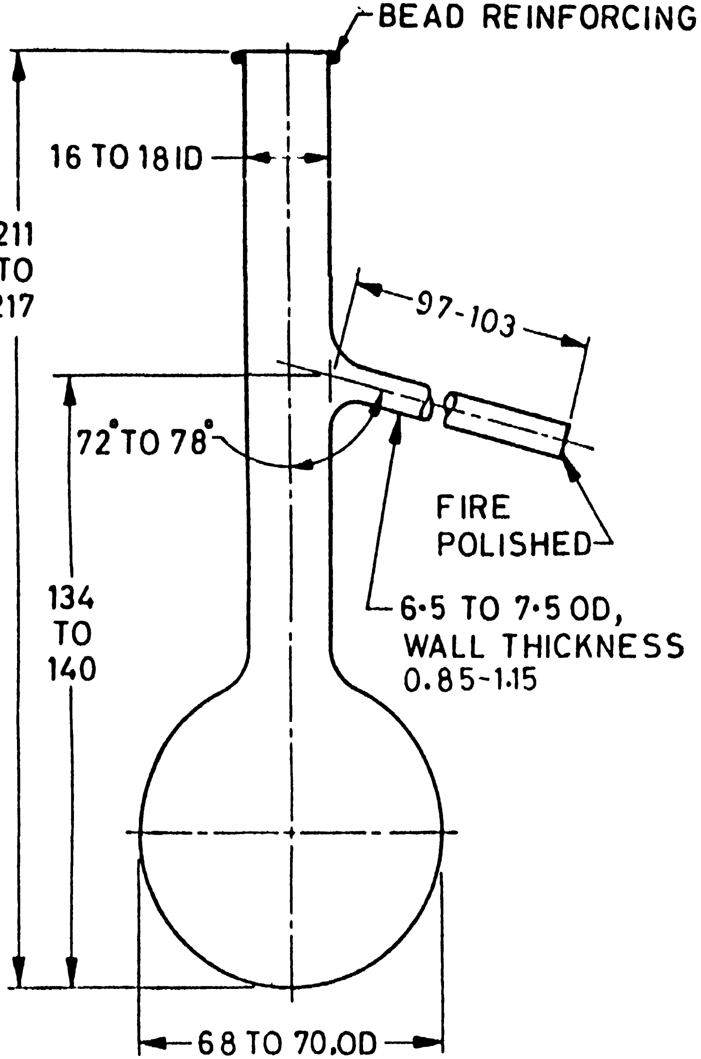
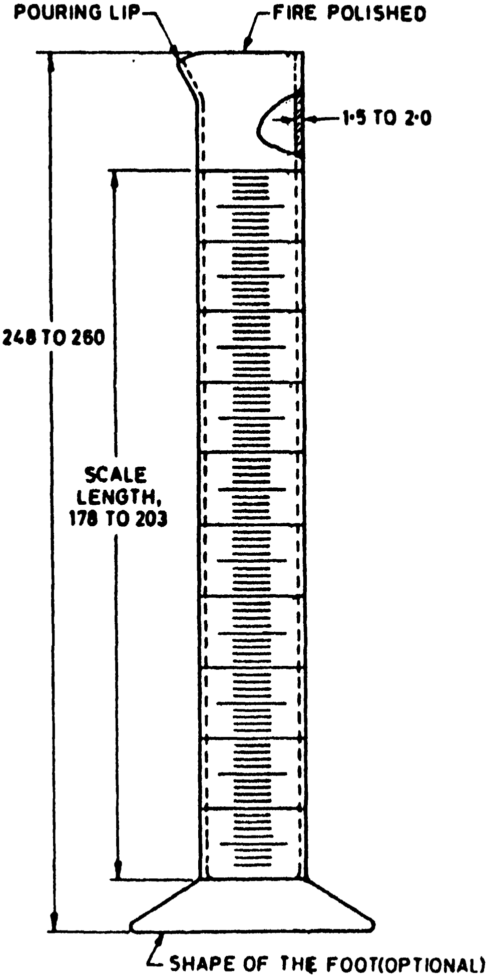


FIG. 5 DISTILLATION FLASK OF 125 ml CAPACITY



NOTES

1 Cylinder must stand on 15° slope without tipping.

2 100 ml in 1 ml graduations.

3 Tolerance ± 1.0 ml.

FIG. 6 GRADUATED CYLINDER

(PCD 1)

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